
ह्यड्रॉज़ाइन हाइड्रेट — विशिष्टि
(पहला पुनरीक्षण)

Hydrazine Hydrate — Specification
(First Revision)

ICS 27.060.30

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FOREWORD

This Indian standard (First Revision) was first published in 1987. This standard covers the requirements and methods of sampling and test for hydrazine hydrate.

This standard is being revised to incorporate method of tests for determination of relative density, residue on evaporation, ash content and ammonia content. In this revision, amendments issued are also incorporated.

The composition of the committee responsible for the formulation of this standard is given at Annex H.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

HYDRAZINE HYDRATE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for hydrazine hydrate.

2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 11671 shall apply.

4 REQUIREMENTS

4.1 The material shall be colourless liquid free from visible impurities and suspended particles.

4.2 The material shall comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in mild steel drums suitably lined with plastic material or in PVC carboys.

5.2 Marking

The containers shall be marked with the following:

- a) Name of the material;
- b) Net volume or mass;
- c) Batch number;
- d) Name of the manufacturer or his registered trade-mark, if any; and
- e) Suitable cautionary note as given below:

NOTE — It has been reported that hydrazine hydrate is likely to have some carcinogenic properties. It is recommended that hydrazine hydrate should not be used in boilers for generation of steam, which will be used in a process industry like processing food and/or skin products.

5.2.1 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

Table 1 Requirements for Hydrazine Hydrate

(Clauses 4.2 and 7.1)

SI No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to (4)
i)	Purity (as hydrazine hydrate), percent by mass, <i>Min</i>	80	Annex B
ii)	Water insoluble matter, percent by mass, <i>Max</i>	0.005	Annex C
iii)	Relative density, 20/20 °C, <i>Min</i>	1.05	Annex D
iv)	pH of 1 percent solution at 20 °C, <i>Min</i>	10.5	IS 3025 (Part 11)
v)	Colour, Hazen units, <i>Max</i>	10	IS 3025 (Part 4)
vi)	Boiling range	115 to 119 °C	IS 5298
vii)	Residue, on evaporation, mg/l, <i>Max</i>	0.01	Annex E
viii)	Ash content, g/100 ml, <i>Max</i>	0.003	Annex F
ix)	Iron (as Fe), ppm, <i>Max</i>	5	IS 3025 (Part 53)
x)	Copper (as Cu), ppm, <i>Max</i>	5	IS 3025 (Part 42)
xi)	Ammonia content (as NH ₃), percent by mass, <i>Max</i>	0.3	Annex G
xii)	Silica (as SiO ₂), ppm, <i>Max</i>	5	IS 3025 (Part 35)
xiii)	Chloride (as Cl), ppm, <i>Max</i>	5	IS 3025 (Part 32)
xiv)	Sulphates (as SO ₄), ppm, <i>Max</i>	5	IS 3025 (Part 24)

6 SAMPLING

6.1 General Requirements of Sampling

6.1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

6.1.2 The sampling instruments shall be clean and dry.

6.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination. To draw a representative Sample from a container, the material shall be mixed thoroughly by suitable means before sampling.

6.1.4 The sample shall be placed in clean and air-tight glass bottles or other suitable containers on which the material has no action and which have been previously washed several times with the material to be sampled.

6.1.5 The sample containers shall be of such a size that they are filled by the sample leaving an ullage of not more than five percent.

6.1.6 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the materials.

6.2 Scale of Sampling

6.2.1 Lot

All containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

6.2.2 For ascertaining conformity of the material in a lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling
(Clause 6.2.2)

Sl No.	Lot Size	Sample Size
	<i>N</i>	<i>n</i>
(1)	(2)	(3)
i)	3 – 15	3
ii)	16 – 40	4
iii)	41 – 65	5
iv)	65 – 110	7
v)	111 and above	10

6.2.2.1 In order to ensure randomness of selection, the following procedure shall be adopted:

‘Arrange all the containers batchwise in the lot in a systematic manner and starting from any one, count them as 1, 2, 3,....., up to *r*, where *r* is the integral part of N/n (*N* and *n* being the lot size and sample size respectively). Every *r*th container thus counted shall be withdrawn to constitute the test sample.’

6.3 Preparation of Test Samples

6.3.1 From each of the containers selected according to 6.2.2.1, equal portions of the material shall be taken out so that the total quantity collected from all the containers is about 3 litre. This shall be the composite sample.

6.3.2 The composite sample shall be divided into 3 test samples not less than 1 litre each. These test samples shall be transferred immediately to clean dry bottles which are sealed air-tight with glass stoppers and marked with the particulars of sampling as given in 6.1.6. One test sample shall be sent to the purchaser and one to the supplier. The third test sample bearing the seals of the purchaser and the supplier shall constitute the referee sample, to be used in case of dispute.

6.3.3 Tests for determination of all characteristics shall be conducted on the composite sample.

6.4 Criteria for Conformity

The lot shall be declared as conforming to the requirements of this specification if all the test results on the composite sample satisfy the corresponding requirements.

7 TESTS

7.1 Test shall be conducted as prescribed in col 4 of Table 1.

7.2 Quality of Reagents — Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities.

ANNEX A

(Clause 2)

LIST OF REFERRED INDIAN STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water (third revision)	3025 (Part 35) : 1988	Methods of sampling and test (physical and chemical) for water and waste water: Part 35 Silica
2263 : 1979	Methods of preparation of indicator solution (first revision)	3025 (Part 42) : 1992	Methods of sampling and test (physical and chemical) for water and waste water: Part 42 Copper
3025 (Part 4) : 1983	Methods of sampling and test (physical and chemical) for water and waste water: Part 4 Colour	3025 (Part 53) : 2003	Methods of sampling and test (physical and chemical) for water and waste water: Part 53 Iron
3025 (Part 11) : 1983	Methods of sampling and test (physical and chemical) for water and wastewater: Part 11 pH value	3025 (Part 54) : 2003	Methods of sampling and test (physical and chemical) for water and waste water: Part 54 Nickel
3025 (Part 24) : 1986	Methods of sampling and test (physical and chemical) for water and wastewater: Part 24 Sulphates	5298 : 2013	Method for determination of distillation range and of distillation yield
3025 (Part 32) : 1988	Methods of sampling and test (physical and chemical) for water and waste water: Part 32 Chloride	11255 (Part 6) : 1999	Methods of measurement of emissions from stationary sources: Part 6 Ammonia.
		11671 : 1985	Glossary of terms relating to boiler water

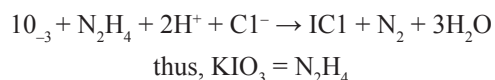
ANNEX B

[Table 1, Sl No. (i)]

DETERMINATION OF PURITY OF HYDRAZINE HYDRATE

B-1 PRINCIPLE

Hydrazine reacts with potassium iodate under the usual Andrew's conditions as follows:



B-2 REAGENTS

B-2.1 Potassium Iodate Solution — 0.025 M. Dissolve exactly 5.35 g of dried potassium iodate in one litre of de-oxygenated water.

B-2.2 Carbon Tetrachloride

B-3 PROCEDURE

Weigh accurately about 0.08 to 0.1g of hydrazine

hydrate. Add a mixture of 30 ml of concentrated hydrochloric acid and 20 ml of deoxygenated water. Shake well. Add 5 ml of carbon tetrachloride. Titrate against potassium iodate solution with continuous shaking. At first the colourless organic layer of carbon tetrachloride changes to blood red and then just decolourises at the end point.

B-4 CALCULATION

$$\text{Purity of hydrazine hydrate, percent} = \frac{0.00125 \times V_1 \times 100}{M}$$

where

V_1 = volume of potassium iodate solution, in ml;
and

M = mass of the sample, in g.

ANNEX C

[Table 1, Sl No. (ii)]

DETERMINATION OF WATER INSOLUBLE MATTER

C-1 PROCEDURE

Weigh accurately about 10 g of the material in a beaker and add equal quantity of water. Mix well with a glass rod. Filter the contents of the beaker quantitatively through a previously weighed sintered glass crucible No. 4 under suction. Wash the beaker with water and filter this also. Similarly wash the crucible a few times with water. Dry the crucible in an air oven at 105 ± 2 °C. Weigh the dried crucible for constant weight.

C-2 CALCULATION

C-2.1 Water insoluble matter, percent by mass =

$$\frac{M_1 \times 100}{M_2}$$

where

M_1 = mass of the residue after drying, in g; and

M_2 = mass of the sample taken for test, in g.

ANNEX D

[Table 1, Sl No. (iii)]

DETERMINATION OF RELATIVE DENSITY

D-1 PRINCIPLE

Determination of density is based on determination of the mass of a known volume of sample at a given temperature.

D-2 APPARATUS

D-2.1 Density Bottle, 50 ml capacity.

D-2.2 Balance, capable of weighing the density bottle to the nearest 0.1 mg.

D-2.3 Water Bath, constant temperature 20 ± 0.5 °C.

D-3 PROCEDURE

Adjust the temperature of the sample to 20 ± 0.5 °C. Fill the tared bottle with sample. Stopper and wipe it. Weigh the bottle the nearest 0.1 mg. If a constant temperature bath is not available, record the temperature with an accurate thermometer and obtain value of relative density for that temperature from Table 3.

D-4 CALCULATION

D-4.1 Calculate the density of the sample as follows:

$$D = \frac{M \times C}{V}$$

where

d = density of the sample at 20 °C, g/ml;

m = mass of the sample in the density bottle, g;

v = Volume of density bottle, ml; and

c = correction factor for temperature

NOTE — When measurements are made at 20 °C, C = 1.
For other temperatures

$$C = \frac{\text{Relative density at 20 °C}}{\text{Relative density at test temperature}}$$

Table 3 Density of Water at Different Temperature

(Clause D-3)

Sl No.	Temperature °C	Density g/ml	Temperature °C	Density g/ml
(1)	(2)	(3)	(4)	(5)
i)	0	0.999 87	50	0.98573
ii)	3.98	1.000 00	55	0.98573
iii)	5	0.999 99	60	0.98324
iv)	10	0.999 73	65	0.98059
v)	15	0.999 17	70	0.97781
vi)	18	0.998 62	75	0.97489
vii)	20	0.998 23	80	0.97183
viii)	30	0.995 67	85	0.96865
ix)	35	0.994 06	90	0.96565
x)	38	0.992 99	95	0.96192
xi)	40	0.992 24	100	0.95838
xii)	45	0.990 25		

NOTE — The temperature of maximum density of pure water, free from air = 3.98 °C.

ANNEX E

[Table 1, Sl No. (vii)]

DETERMINATION OF RESIDUE ON EVAPORATION**E-1 PRINCIPLE**

The volume of the sample is allowed to evaporate to dryness and the weight of the residue is noted.

E-2 APPARATUS

E-2.1 Platinum Dish, 100 ml capacity or Nickel/Silica dish.

E-2.2 Pipette, 100 ml.

E-2.3 Steam Bath, Constant temperature.

E-2.4 Hot Air Oven, calibrated for 103 °C.

E-2.5 Dessicator

E-2.6 Balance capable of weighing the density bottle to the nearest 0.1 mg.

E-3 PROCEDURE

E-3.1 Heat a clean platinum dish of about 100 ml capacity to redness and cool it in dessicator. Weigh the

dish. Alternatively, a nickel or silica dish may be used in which case dry it at about 105 °C for 30 min and then cool it to room temperature.

E-3.2 Pipette out 100 ml of the well mixed sample in stages into the weighed dish, and evaporate to dryness on a steam bath. Wipe the outside of the dish and dry the residue for one hour at 103 to 105 °C. Transfer the dish to a dessicator and weigh it as soon as room temperature is reached. Repeat drying and weighing till the weight is constant to within 0.5 mg. Reserve the residue for test in **F-3.1**. Express the result to nearest 5 mg/l.

E-4 CALCULATION

$$\text{Residue content, mg/l} = \frac{W \times 10^6}{v}$$

where

W = weight of residue obtained, in g and

v = volume of the sample taken, in ml.

ANNEX F

[Table 1, Sl No. (viii)]

DETERMINATION OF ASH CONTENT

F-1 PRINCIPLE

F-1.1 Incineration of the Dried Residue Results the Ash Content.

F-2 APPARATUS

F-2.1 Dessicator

F-2.2 Furnace, calibrated for 525 to 550 °C.

F-3 PROCEDURE

F-3.1 Ignite the residue reserved in E-3.2 at 525 °C

to 550 °C for 30 min. Weigh the ignited residue after cooling it in a dessicator to room temperature.

NOTE — If there is any odour or change of colour during ignition, include it in the report.

F-3.2 Calculation

$$\text{Ash, g/100 ml} = \frac{W}{v}$$

where

W = weight of the ignited residue, in g and

v = volume of the sample taken in E-3.2, in ml.

ANNEX G

[Table 1, Sl No. (xi)]

DETERMINATION OF AMMONIA CONTENT

G-1 Ammonia may be determined by either of the two methods, namely, Method A and Method B. In case of dispute Method B shall be adopted.

G-1.1 Method A

G-1.1.1 Reagents

G-1.1.1.1 Standard sulphuric acid — 0.5 N

G-1.1.1.2 Methyl red indicator — Dissolve 0.5 g of water soluble methyl red in water and dilute the solution to one litre.

G-1.1.1.3 Caustic soda solution — 0.5 N, freshly standardized.

G-1.1.2 Procedure

Accurately weigh a 15 ml capacity ground glass stoppered weighing bottle containing 10 to 12 ml of water. Open the container of hydrazine hydrate and insert a clean and dry glass tube of about 5 mm bore to half the height of the liquid and transfer 1 to 1.5 ml of the material to the weighing bottle, immediately close the glass stopper and weigh. Place the weighing bottle in about 200 ml of water containing 50 ml of 0.5 N sulphuric acid and a few drops of methyl red indicator.

The bottle will be immersed in acid due to mass of water and sample in it. Open the stopper under the acid by manipulating with a glass rod and allow to react the content of weighing bottle with acidified solution. Titrate the excess of acid with standardized caustic soda solution to faint yellow end point.

G-1.1.2.1 Carry out a blank with all the reagents used for the test except sample.

G-1.1.3 Calculation

$$\text{Ammonia, percent by mass} = \frac{1.703 \times (V_1 - V_2) \times N}{M}$$

where

V_1 = Volume of standard sodium hydroxide solution used in blank, in ml

V_2 = volume of standard sodium hydroxide solution used in the test with the material, in ml

N = normality of standard sodium hydroxide solution, and

M = mass of the material taken for the test, in g.

G-1.2 Method B

G-1.2.1 Reagents

G-1.2.1.1 Boric acid — 2 percent.

G-1.2.1.2 Standard sulphuric acid solution — 0.5 N.

G-1.2.1.3 Methyl red — 0.1 percent. Dissolve 0.1 g of methyl red in 95 percent (v/v) ethanol and make up to 100 ml with the same ethanol.

G-1.2.2.1 Spherical glass ampoule-of suitable capacity and shape, for example, about 20 mm diameter, with one capillary end about 50 mm in length (a typical example is shown in Fig. 1).

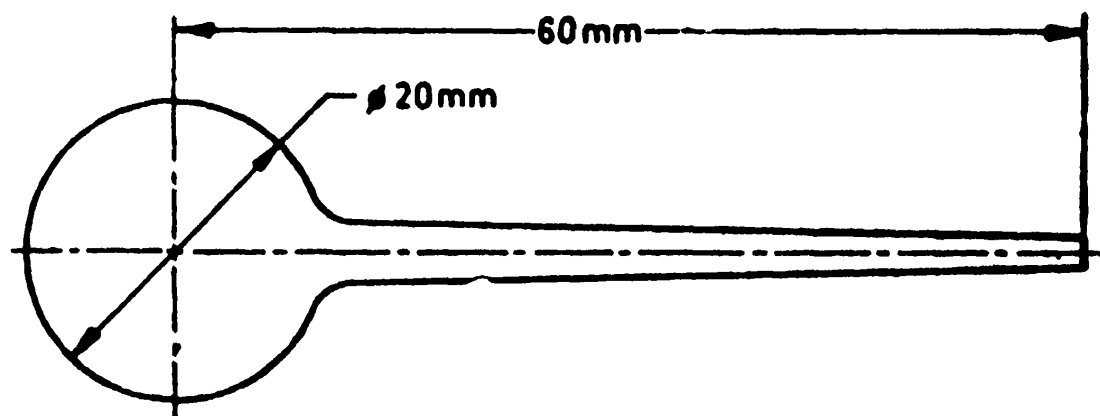


FIG. 1 SPHERICAL GLASS AMPOULE

G-1.2.3 Procedure

Weigh the glass ampoule to the nearest 0.01g. Gently heat the spherical part of the ampoule over a flame and dip the capillary end of the ampoule into the bottle containing the laboratory sample. Ensure that the ampoule is almost completely filled during cooling.

Withdraw the ampoule and dry the capillary tube carefully with filter paper. Seal the end of the capillary tube, without loss of glass, with an oxidizing flame. Allow the capillary tube to cool, wash with water and wipe it carefully with filter paper.

Weigh the sealed ampoule accurately and calculate, by difference, the mass of the sample in it.

G-1.2.3.2 Carefully place the ampoule containing the portion into a 500 ml conical flask with a ground glass stopper, to which 50 ml of the boric acid solution, about 250 ml of water and several drops of the methyl red solution have already been added.

Stopper the conical flask and shake carefully so as to break the ampoule inside the flask.

Unstopper the flask, rinse the stopper with water collecting the washings in the same flask.

Using a glass rod, grind the pieces of the ampoule, in particular those parts of the capillary tube which may have remained unbroken. Remove the glass rod, rinse it with water, collecting the washing in the same flask.

Titrate with the sulphuric acid solution until the indicator changes from yellow to red.

G-1.2.4 Calculation

$$\text{Ammonia, percent by mass} = \frac{1.703 \times V}{M}$$

where

V = volume of standard sulphuric acid used, in ml
and

M = mass of the material taken for the test, in g.

ANNEX H

(Foreword)

COMMITTEE COMPOSITION

Water Quality for industrial purposes Sectional Committee, CHD 13

<i>Organization</i>	<i>Representative(s)</i>
Bhabha Atomic Research Centre, Mumbai	SHRI K. P. BHATTACHARYYA (Chairman) SHRI SHIVAYANAMATH. S (<i>Alternate</i>)
Bharat Heavy Electrical Limited, Thiruchirapali	DR S. BHATNAGAR K. ANANDA BABU (<i>Alternate</i>)
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Central Salt and Marine Chemicals Research Institute, Bhavnagar	DR SOUMYA HALDAR DR D. N. SRIVASTAVA (<i>Alternate</i>)
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National Environment Engineering Research Institute, Nagpur	DR PAWAN LABHASETWAR ER S. P. ANDEY (<i>Alternate</i>)
NLC Nalco India Limited, Kolkata	DR ASHISH KUMAR SAMADDAR SHRI NADAN CHATTERJI (<i>Alternate</i>)
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Padarsh Pharmaceuticals Private Limited, Mumbai	SHRI MUNEESH CHADDA SHRI S. Y. PANDIT (<i>Alternate</i>)
Shriram Institute for Industrial Research, Delhi	DR V. K. VERMA MS MANJEET AGGARWAL (<i>Alternate</i>)

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The Fertilizer Association of India, New Delhi	SHRI MANISH GOSWAMI SHRI N. R. KAMAT (<i>Alternate</i>)
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Member Secretary

SHRI SAGAR SINGH
SCIENTIST 'C' (CHD), BIS

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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